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Alan Arias

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Alan Arias Lewis Research Center Cleveland, Obio



Scientific and Technical Information Branch

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Summary

The main objective of the present investigation was to determine the effect of yttria (Y_2O_3) additions on the modulus of rupture (MOR) and oxidation resistance of pressureless-sintered silicon nitride (Si_3N_4) prepared from milled Si_3N_4 powders contaminated with 5.9 wt% oxygen (11.1 wt% silicon dioxide (SiO_2)). A secondary objective was to determine the effect of substituting alumina $(A1_2O_3)$ for Y_2O_3 in one of the compositions investigated.

Cold-pressed bars were sintered at 1760° C for 4 hours in static nitrogen at 34.5-MPa (5-psi) gage pressure while embedded in a mixture of coarse Si₃N₄+5 wt% SiO₂ powders. Ground bars of compositions containing 3.67, 7.22, and 14.0 wt% Y₂O₃ had average MOR's of 460, 515, and 515 MPa at room-temperature and 270, 256, and 227 MPa at 1400° C, respectively. A material with 3.74 wt% $Y_2O_3 + 1.69$ wt% $A1_2O_3$ had average MOR's of 452 and 118 MPa at room temperature and 1400° C, respectively. The corresponding MOR's of the best pressureless-sintered Si₃N₄-Y₂O₃ or Si₃N₄-Y₂O₃-Al₂O₃ materials reported in the literature are up to 50 percent higher than these. On the other hand the oxidation resistance of the $Si_3N_4-Y_2O_3$ materials from the present investigation was much better than that of the same high-strength materials as well as of hot-pressed Si₃N₄. At all temperatures investigated (600° to 1400° C) the oxidation resistance of the Si₃N₄-SiO₂-Y₂O₃ compositions decreased with increasing Y_2O_3 . Also, although up to ~1200° C the addition of Al₂O₃ did not appear to have much effect on oxidation resistance, it had a very detrimental effect at 1400° C. It is surmised that the lower high-temperature strength and higher oxidation resistance of the Si₃N₄-SiO₂-Y₂O₃ materials from the present investigation (as compared with values in the literature) were due to their higher SiO₂ content. None of the compositions investigated showed cracks, blisters, or discolorations, such as reported in the literature for some Si₃N₄-Y₂O₃ compositions, during oxidation in air in the 600° to ~1000° C range.

Introduction

In a previous NASA investigation (ref. 1) it was shown that high-density Si₃N₄ base ceramics could be

made by pressureless sintering milled Si_3N_4 powders with additions of cerium oxide (CeO₂), magnesium oxide (MgO), or yttrium oxide (Y₂O₃). Although all three additives or their mixtures yielded sintered specimens with about the same maximum percent of theoretical density, it was surmised that Y₂O₃ additions were probably best for optimization of high-temperature properties.

The main objective of the present investigation was to determine the effect of Y2O3 additions on the modulus of rupture (MOR) and oxidation resistance of pressureless-sintered Si₃N₄ prepared from milled powders. A secondary objective was to determine the effect of substituting Al₂O₃ for Y₂O₃ in one of the compositions investigated. To attain these objectives, compositions were made from milled Si₃N₄ powders and three different levels of Y2O3. In addition, a fourth composition containing both Y2O3 and Al2O3 as sintering aids was prepared for comparison. These four compositions were made essentially by the methods of reference 1, which involved the pressureless sintering of prismatic bars made by cold pressing. The sintered and surface-ground bars were tested for MOR and oxidation resistance as functions of temperature.

Materials, Equipment, and Procedure

The materials used in the present investigation were powdered α -Si₃N₄, Y₂O₃, and Al₂O₃. These materials in the "ready for compounding" condition are characterized in table I.

The equipment used in this investigation is described at length in reference 2. Briefly, it consisted of 1.5-liter-capacity, nickel-lined ball mills with nickel shot as the grinding medium and standard laboratory equipment (presses, dies, furnaces, etc.).

The procedures used for the preparation and testing of bars are outlined in the flow diagram of figure 1. Since the procedures used differed only slightly from those used in the work of reference 1, they are described only briefly in this section.

The α -Si₃N₄ was milled with water for 300 hours. During the milling operation the powder picked up nickel from balls and mills. Most of this nickel was removed from the Si₃N₄-water slurry magnetically. The rest of the nickel was removed from the slurry by leaching with nitric acid (HNO₃), centrifuging to remove most of the water, washing the moist

TABLE I, - CHARACTERIZATION OF RAW MATERIALS

Material	Source;	Milling data				Post-	Specific surface	Chemical analysis			
	designation	Mill material	Mill Balls Milling Milling treatment are atternal fluid time, pov		area of powder, m ² /g	Oxygen, percent	Carbon, percent	Other elements, ppm unless noted otherwise (spectrographic analyses)			
α-Si ₃ N ₄	Kawecki-Berylco Industries; HP	Ni	Ņi	Water	300	Leach, centrifuge, wash, cen- trifuge, dry	22.0	5, 9	0.14 a _{0.48}	0, 3%Al-90Ca-60Cr- 40Cu-0, 2%Fe-50Mg- 20Mn-300Ni-Si major- 70Ti-70Zr	
Al ₂ O ₃	Union Carbide Corp.; Linde A				0		14, 5	(b)	0, 10	80 Ca-<10 Co-5 Cr- <50 Cu-20 Fe-<5 Mn- <20 Mo-<100 Nb-<5 Ni- 50 Pb-50 Sn-20 Ti- <10 V-<40 W-<100 Y- <10 Zr	
Y ₂ O ₃	Research Chemicals; 99.9 percent pure				0		69. 9	(b)	0.14	50Al-75Ca-<10Co-2Cr- <50Cu-<20Fe-<5Mn- <20Mo-2000Na-<100Nb- 5Ni-<100Pb-<50Sn- 5Ti-<10V-<40W-Y major-<10Zr	

^aThis carbon content obtained after mixing 1 hr with 5 wt% silicone oil and 70 wt% ethanol, then heating to 450° C for 1 hr in flowing nitrogen.
bNot determined.

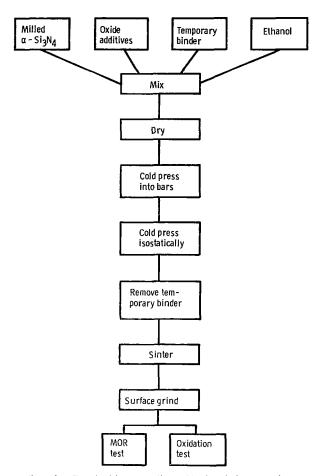


Figure 1. - Flow chart for preparation and testing of Si₃N₄ ceramics.

powder, centrifuging to remove the last traces of dissolved nickel nitrate $(Ni(NO_3)_2)$ and HNO_3 , drying the moist powder cake, and finally pulverizing the dry powder agglomerates in a Waring Blendor. The milled Si_3N_4 , as well as the as-received and dried Y_2O_3 and Al_2O_3 powders, was analyzed for oxygen and carbon and spectrographically analyzed for trace elements. The specific surface areas of these powders were determined by the BET (Brunauer, Emmett, and Teller) method.

The Y₂O₃ and A1₂O₃ were used in the as-received and dried condition. Because a temporary binder (DC-705 silicone oil) and ethanol were used for specimen preparation, some carbon residue remained in the powder compacts after removal of the binder. This carbon residue was determined by separate experiments, and its value (which was about the same in all the compacted compositions) is shown in table I.

Calculated amounts of the powders characterized in table I were weighed in an analytical balance and mixed with 5 wt% temporary binder and about 70 wt % absolute ethanol. The powders were mixed for 1 hour in a polyethylene bottle with the aid of a few stainless-steel balls. The slurry was dried at about 100° C, and the resulting agglomerates were pulverized in a Waring Blendor. The powder mixtures were shaped into bars approximately 3.81 by 0.92 by 0.47 centimeter by cold pressing at 207 MPa (30 ksi) in a double-acting steel die. These bars were then isostatically cold pressed at 483 MPa (70 ksi). The silicone oil used as temporary binder was removed from the bars by heating them slowly (for ~2 hr) in flowing nitrogen to 450° C and holding for 1 hour.

The bars to be sintered were placed in a graphite sintering boat and packed all around with a mixture of -325 mesh α -Si₃N₄ (Kawecki-Berylco, CP 85) and 5 wt% -325 mesh SiO₂ (Cerac Pure, C-1064). Sintering was carried out at 1760° C for 4 hours in a static nitrogen atmosphere held at 34.5-MPa (5-psi) gage pressure.

The sintered bars were surface ground into 2.54-by 0.635- by 0.318-cm test bars. The bars to be used for MOR tests had their edges beveled 0.12 mm and were tested in the as-ground condition. The bars to be used for oxidation tests were polished on all sides to a mirror finish on a $10-\mu m$ diamond lap.

The four-point MOR was determined at various temperatures from room temperature to 1400° C. A silicon carbide fixture having a 1.905-cm (3/4-in.) bottom span and a 0.953-cm (3/8-in.) top span was used for this purpose. The test bar and fixture were heated to the test temperature in air in a silicon carbide muffle furnace mounted on an Instron tensile tester. The bars were bend tested at a crosshead speed of 0.051 cm/min (0.020 in/min).

Oxidation tests in air were carried out at temperatures from 600° to 1400° C. During these tests the bars were held in an alumina crucible between alumina wedges. To allow access of air, the crucible had two side holes. At various times the bars were cooled, removed from the crucible, and weighed in an analytical balance with microgram sensitivity in order to determine the cumulative weight gains as a function of time.

Results and Discussion

The compositions used in the present investigation are listed in table II. The Si_3N_4 used for making these compositions contained 11.1 wt% SiO_2 , as contrasted with Si_3N_4 with 14.5 wt% SiO_2 used for making some compositions containing the same amounts of Y_2O_3 in reference 1. Therefore, except

TABLE IL - COMPOSITIONS INVESTIGATED

Designationa	Composition							
	Si ₃ N ₄		Sic	02 ^b	$^{\mathrm{Y}_{2}\mathrm{O}_{3}}$		Al ₂ O ₃	
	wt%	eq%	wt%	eq%	wt%	eq%	wt%	eq%
A	8 5. 64	90.05	10, 69	8. 75	3, 67	1, 20		
В	82. 48	88.94	10. 29	8, 64	7. 22	2. 42		
С	76, 48	86, 66	9, 54	8. 42	14.0	4, 92		
Dc	84. 07	88.91	10, 49	8, 63	3, 74	1, 23	1. 69	1. 23

^aCompositions A, B, and C are equivalent to Y2, Y4, and Y8, respectively, in ref. 1.

for having somewhat lower SiO_2 , compositions A, B, and C, which contain 3.67, 7.22, and 14.0 wt% Y_2O_3 are equivalent to Y2, Y4, and Y8, respectively, in reference 1. In addition, X-ray diffraction analyses showed the same phases (β -Si₃N₄+Si₂N₂O) in the sintered ground bars. Composition D in table II is similar to composition B except that half the Y_2O_3 was replaced with $A1_2O_3$ on a mole or equivalent percentage basis. This was done for comparison.

The densities of sintered and ground bars of compositions A, B, C, and D were 97.2, 97.8, 96.0, and 96.8 percent of theoretical, respectively. These theoretical densities were calculated on the basis of the densities of hot-pressed bars, after adjustments for compositional differences and as described in reference 1.

Modulus of Rupture

The four-point MOR's of compositions A, B, and C (table II) as functions of temperature are shown in figure 2. For comparison the four-point-average MOR of GTE Si_3N_4+6 wt% Y_2O_3 from reference 3 is also shown in this figure. As the plots in figure 2 show, the room-temperature MOR's of compositions A, B, and C ranged from 416 to 594 MPa, decreasing gradually with increasing temperature to the range of 207 to 312 MPa at 1400° C. The plots also show that

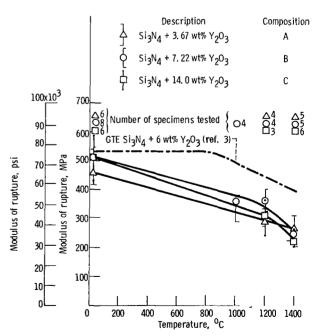


Figure 2. - Effect of temperature and Y₂O₃ additions on fourpoint modulus of rupture of pressureless-sintered Si₃N₄.

bCalculated from the oxygen in the milled Si₃N₄ (table I).

^cComposition D is similar to composition B, except that half the Y₂O₃ was replaced with Al₂O₃, on a mole or equivalent percentage basis.

composition A (3.67 wt% Y₂O₃) had an average MOR that was lower at room temperature but slightly higher at 1400° C than the average MOR's of compositions B and C, which had a higher Y₂O₃ content. It is clear from the plots in figure 2 that at 1400° C the MOR decreased with increasing Y₂O₃ content. On the other hand, at room temperature the compositions with the higher Y₂O₂ contents (B and C) were stronger, but it is not clear whether this increased strength was brought about by the higher Y₂O₂ content or by some other unknown factor. Comparison of the plots for compositions B and C with that for the GTE Si₃N₄+6 wt% Y₂O₃ shows them to have comparable strength at room temperature, but the GTE material was about 50 percent stronger at 1400° C. The higher hightemperature strength of the GTE material was probably due to its lower (~5.4 wt%, ref. 3) SiO₂ content and correspondingly smaller amounts of glass phase. Because of its lower SiO2 content the GTE material was made up of β -Si₃N₄+ $Y_2Si_2O_7 + 10Y_2O_3 \cdot 9SiO_2 \cdot Si_3N_4$; and this phase composition, as is shown later, does not have as good oxidation resistance as β -Si₃N₄ + Si₂N₂O.

In figure 3 are shown plots of the four-point MOR's as functions of temperature for compositions C (7.22 wt% Y_2O_3) and D (3.74 wt% $Y_2O_3 + 1.69$ wt% $A1_2O_3$). For comparison, plots of the MOR's as functions of temperature for GTE $Si_3N_4 + 6$ wt%

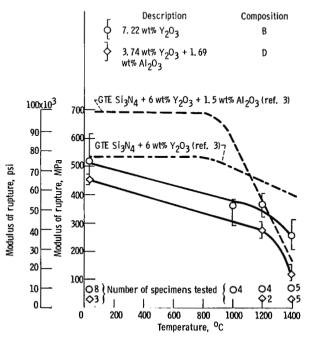


Figure 3. - Effect of temperature and additions of Y₂O₃ or Y₂O₃ + Al₂O₃ on four-point modulus of rupture of pressure-less-sintered Si₃N₄.

 Y_2O_3 and GTE Si_3N_4+6 wt% $Y_2O_3+1.5$ wt% Al₂O₂ are also included in this figure. As the plots show, of the compositions from the present investigation, the one with Al₂O₃ had lower strength at all temperatures than the one without it. Also. although substitution of Al₂O₃ for half the Y₂O₃ moles decreased the strength at room temperature by about 12 percent, it decreased the strength at 1400° C by about 50 percent. On the other hand, of the GTE materials in figure 3, the one with Al₂O₃ was about 22 percent stronger at room temperature and about 50 percent weaker at 1400° C. From these comparisons it is surmised that Al₂O₃ is detrimental to the high-temperature strength of Si₃N₄-SiO₂-Y₂O₃ compositions. This holds true whether the Al₂O₃ in material D was regarded as a substitution for Y₂O₃ in composition B or as an addition to the Y₂O₃ in composition A, since the room-temperature strengths of compositions A, B, and D were (within the limits of experimental error) just about the same, as comparison of the plots in figures 2 and 3 shows. On the other hand, it is not clear why Al₂O₃ (whether regarded as an addition to, or substitution for, Y₂O₃) improved the roomtemperature strength of the GTE material but not of the materials (either A or B) from the present investigation. This difference in room-temperature strength may be due to

- (1) Different amounts of glass phase
- (2) Different grain sizes (unknown)
- (3) Different impurity contents (the GTE material being much purer)
- (4) Different kinds and amounts of crystalline phases
- (5) Different porosity (96.8 percent of theoretical density for composition D as compared with 100 percent for the GTE material)
- (6) A combination of these factors

Oxidation

The weight increases with time on oxidizing compositions A, B, and C (table II) in air at temperatures from 1000° to 1400° C are plotted in figure 4. For comparison, 1400° C oxidation data for hot-pressed Si_3N_4 (ref. 4) and 1000° C oxidation data for pressureless-sintered GTE Si_3N_4+6 wt% Y_2O_3 (ref. 3) are also plotted in figure 4. Comparison of the plots from the present investigation shows that, at a given temperature and time at temperature, weight gains increased with increasing Y_2O_3 . From this it is concluded that Y_2O_3 additions are detrimental to oxidation resistance. Yet, even the composition with 14 wt% Y_2O_3 had better 1400° C oxidation resistance than the hot-pressed, commercial Si_3N_4 . Although 1400° C oxidation data

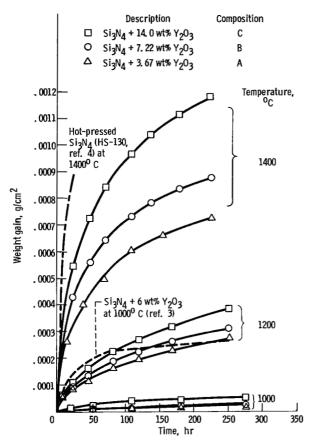


Figure 4. – Weight gain of Si_3N_4 – Y_2O_3 compositions oxidized in air at temperatures indicated.

for the GTE material are not presently available, the 1000° C data from reference 3 presented in figure 4 show that the GTE material had a much higher weight gain than materials from the present investigation with equivalent amounts of Y2O3. The better oxidation resistance of the materials from the present investigation is attributed to their higher SiO₂ content. This SiO₂ content is such (table II) that even with up to 14.0 wt% Y₂O₃ the compositions from the investigation fall within Si₃N₄-Si₂N₂O-Y₂Si₂O₇ compatibility triangle of the Si₃N₄-SiO₂-Y₂O₃ phase diagram and should, according to reference 5, have better oxidation resistance.

The increase in weight with time on oxidizing composition D (Si₃N₄+3.74 wt% Y₂O₃+1.69 wt% A1₂O₃) in air at temperatures from 600° to 1400° C is plotted in figure 5. For comparison, the corresponding plots for hot-pressed (HP) Si₃N₄ at 1400° C (ref. 4) and for GTE Si₃N₄+4 wt% Y₂O₃+2 wt% A1₂O₃ at 1350°, 1200°, and 1000° C (ref. 3) are also plotted in figure 5. Comparison of the plots for composition D in figure 5 with those for

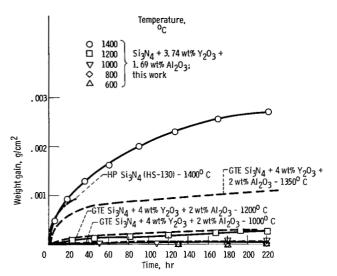


Figure 5. – Comparison of weight gain of Si_3N_4 with 3. 74 wt% Y_2O_3 + 1. 69 wt% Al_2O_3 oxidized in air at temperatures from 600° to 1400° C to weight gain of other Si_3N_4 materials.

composition B in figure 4 shows that replacement of Y₂O₃ with Al₂O₃ markedly reduced the oxidation resistance of $Si_3N_4 + 7.22$ wt% Y_2O_3 at 1400° C but that at 1200° and 1000° C the replacement of Y₂O₃ with Al₂O₃ had no significant effect on oxidation. Thus, for instance, after oxidation in air for 220 hours compositions B and D gained in weight 8.7×10^{-4} and 27.2×10^{-4} g/cm² at 1400° C, as compared with 3.02×10^{-4} and 2.47×10^{-4} g/cm² at 1200° C, respectively. Figure 5 also shows that at 1000° and 1200° C the $Si_3N_4-SiO_2-Y_2O_3-A1_2O_3$ from both the present investigation and reference 3 had about the same oxidation resistance. In addition, considering the weight gain of the GTE material at 1350° C and the pronounced effect of temperature on weight gain in this temperature range, it is surmised that these two materials also have about the same oxidation resistance at 1400° C. This oxidation resistance is comparable to that of hot-pressed Si₃N₄ at 1400° C (ref. 4).

In addition to the results discussed above, compositions A, B, and C were also oxidized in air at 600° and 800° C for up to 400 hours. At 800° C the increases in weight after 400 hours were ≤ 0.00003 g/cm², and at 600° C the increases in weight were not measurable with the equipment used.

The main conclusion derived from these results is that for the materials from the present investigation replacement of Y_2O_3 with $A1_2O_3$ in Si_3N_4 - SiO_2 base compositions is detrimental to the high-temperature (~1400° C) oxidation resistance. On the other hand, from the data in figure 4, it is surmised that the difference in oxidation resistance at 1000° C between GTE Si_3N_4+6 wt% Y_2O_3 and composition B

probably results from differences in the SiO₂ content of the two types of material, as evidenced by the fact that (as shown in ref. 3) preoxidizing this GTE material at 1350° C markedly decreased the subsequent oxidation rate at 1000° C. This effect is reportedly due to an increase in the concentration of surface SiO₂ (ref. 3). The same effect had previously been reported in reference 5 for Si₃N₄-SiO₂-Y₂O₃ materials with very low SiO₂ content. In this same reference it is reported that certain Si₃N₄-SiO₂-Y₂O₃ compositions are susceptible to degradation (excessive oxidation and cracking) at relatively low temperatures ($\sim 1000^{\circ}$ C). This degradation has been attributed to the phases present (ref. 5), to carbon (ref. 6), and to either the tungsten or tungsten carbide picked up on milling (ref. 7). In this context, it may be of interest to note that none of the samples from the present investigation exhibited cracking, spalling, or any other abnormal effect on oxidizing them in air at any temperature from 600° to 1400° C.

The results of the present investigation also show that, at least for Si_3N_4 with about 6 wt% oxygen (~11 wt% SiO_2), $A1_2O_3$ is detrimental to high-temperature strength and high-temperature oxidation resistance without having much effect on sinterability. Considering the results of Smith et al. with their high-purity Si_3N_4 , it is surmised that the approximately 0.6 wt% $A1_2O_3$ in the milled Si_3N_4 used in the present investigation may significantly reduce high-temperature strength. Therefore the use of higher purity Si_3N_4 in compositions made by the methods of the present investigation may well provide Si_3N_4 - SiO_2 - Y_2O_3 compositions with desirable combinations of high-temperature strength and oxidation resistance.

Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio, May 1, 1981

Concluding Remarks

From the results of the present investigation it is concluded that increasing amounts of Y_2O_3 decrease the high-temperature strength and the oxidation resistance of Si_3N_4 - SiO_2 - Y_2O_3 compositions made from milled Si_3N_4 (contaminated with 11.1 wt% SiO_2) containing from 3.67 to 14.0 wt% Y_2O_3 .

The results of the present investigation when compared with data in the literature suggest that SiO₂ is important to the properties of Si₃N₄-base ceramics. This comparison shows that increasing the SiO₂ to suitable levels increases oxidation resistance but decreases high-temperature strength. Therefore, within the constraints imposed by composition and particle size on sinterability, it should be possible to obtain Si₃N₄ base materials with either high strength and low oxidation resistance or vice versa by changing the SiO₂ content of the sintered compositions. This SiO₂ content can be varied by varying the surface area (e.g., by varying milling time) of the starting Si₃N₄ powder, by adding SiO₂, or by removing SiO₂ with carbon additions.

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16. Abstract						
Si ₃ N ₄ -base ceramics were mad additives by pressureless sinter	e from milled Si ring at 1760 ⁰ C.	3N ₄ containing 11.1 The four-point-ave	$1 \ m wt\% \ SiO_2$ and overage moduli of	xide rupture		
were 460, 515, and 515 MPa at	room temperatu	re and 270, 256, an	nd 227 MPa at 14	100 ⁰ С		
for compositions with 3.67, 7.2 sistance of these compositions of	2, and 14.0 wt% decreased with in	Y2O3, respectively acreasing Y2O2 in t	y. The oxidation he 600 ⁰ to 1400 ⁰	n re- C		
range, and no surface oxide cra	cking or spalling	g was noted. Parti:	al substitution of	f Al ₂ O ₃		
for Y ₂ O ₃ reduced both strength	and oxidation re	sistance.				
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